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Development of A Calorimeter for Simultaneously Measuring Heat Release and Mass Loss Rates

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
National Engineering Laboratory
Center for Fire Research
Washington, DC 20234

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**DEVELOPMENT OF A CALORIMETER FOR
SIMULTANEOUSLY MEASURING HEAT
RELEASE AND MASS LOSS RATES**

John Tordella and William H. Twilley

U.S. DEPARTMENT OF COMMERCE
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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, *Secretary*
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director*

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DEVELOPMENT OF A CALORIMETER FOR SIMULTANEOUSLY
MEASURING HEAT RELEASE AND MASS LOSS RATES

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Abstract

A heat release rate calorimeter (designated as NBS II) was designed, built, and put into operation. Specimens may be burned vertically or horizontally without the use of reflectors to provide a uniform external radiant flux field. The flux range is 25 to 80 kW/m². Heat release rate, mass loss rate, smoke, and heat of combustion of the unburned gaseous decomposition products are measured. The heat release rate measurement involves the use of a substitution burner technique which provides fast response. The ratio of the heat release to mass loss rates, the apparent heat of combustion, is provided as a function of time. The calorimeter may also be operated in other modes, e.g., stack temperature increase and oxygen depletion, to obtain the heat release rate.

Representative data are reported for wood and a number of synthetic polymers burned horizontally at a flux of 50 kW/m². These specimens generate considerable heat and a significant amount of uncombusted fuel.

A program involving thorough characterization of the instrument, obtaining reference heat release data for numerous materials, and conducting research in heat release rate and other flammability characteristics is recommended.

Key words: calorimeter; fire test; heat of combustion; heat release rate; mass loss rate.

1. INTRODUCTION

The rate of heat release by materials as they burn is recognized as an important factor governing the rate of fire growth in real world situations involving these materials. A number of devices have been used over the past 10 to 15 years to measure heat release rate [1]¹. Notwithstanding, there was need for an instrument which would:

- (1) accept sufficiently large specimens;
- (2) operate equally well with either vertical or horizontal specimen orientation;

* This work was conducted while Dr. Tordella was a Research Associate at the National Bureau of Standards.

¹ Numbers in brackets refer to literature references listed at the end of this report.

(3) provide rate of mass loss as well as rate of heat release; and,

(4) provide reliable data in a reasonable time.

Existing instruments were deficient in one or more of the above requirements. The Factory Mutual calorimeter developed by Tewarson et al. [2] employs rather small specimens oriented horizontally. The Ohio State University (OSU) calorimeter [3] does not measure mass loss, has slow response, and uses small specimens. The OSU use of a reflector for horizontal specimen exposure with its corresponding shortcomings of smoke dulling and strong reradiative effects introduce several errors. The existing National Bureau of Standards instrument, NBS I [4], employs a relatively small specimen, does not provide for specimen mass loss, and has a poor geometry for horizontal specimens.

A new instrument was designed and built to overcome these deficiencies and satisfy the objectives set forth above. This task was begun in February 1976. Details of the design, construction, and operation are given herein, as are illustrative heat release data and a recommended program for future research.

2. DESIGN

2.1 Specifications

The design specifications for the new National Bureau of Standards heat release rate calorimeter, NBS II, are given in Table 1. These specifications were set by consensus of those at the Bureau who worked or had been involved in heat release rate calorimetry. The design specifications are those for a "state-of-the-art" heat release rate calorimeter.

2.2 Choices

2.2.1 Specimen Size

Specimen sizes of 300 x 300 mm (1 x 1 ft) for vertical specimens and 150 x 300 mm (1/2 x 1 ft) for horizontal specimens were chosen. These sizes were expected to be sufficiently large that the data would be largely independent of specimen size. Specimen area to thickness ratios were anticipated to be large enough that errors due to "edge effects" would be relatively small.

2.2.2 Radiant Panel Type

Gas-heated radiant panels were chosen to provide the desired 80 kW/m² flux. Electric, nichrome resistance heaters do not provide sufficiently high flux for reasonable rates of electrical power consumption. High color temperature, short wavelength radiation electric heaters were avoided because light and dark specimens have different absorptivity at these wavelengths. Gas heated radiant panels with their longer wavelength radiation avoid problems of differential absorption and more nearly represent real world fire exposures. Reflectors, usually used with high color temperature sources, are also a problem. Loss of reflectivity due to coating by smoke particles may reduce the energy reflected to the specimen during a test. Silicon carbide rod heaters, which are now used in the OSU calorimeter, were not considered. These heaters are limited area sources. Decisions between the desirability of these relative to gas panels may be best arrived at through experience.

Radiant panels manufactured by Eclipse Combustion Engineering, Inc.² were chosen over the often used British-made ones. The American panels had the advantages of mounting and general consulting services available for associated equipment supplied by the manufacturer.

2.2.3 Panel Configuration

To provide sufficiently uniform flux for horizontal specimens, the radiant panel heaters were arranged to form the four sides of a box. The horizontal specimen is located at the bottom of this box geometry. For testing vertical specimens, a holder is placed upright at the center of the heated area with the specimen "looking into" the radiant panels.

The arrangement of the radiant panels is shown schematically in figures 1 and 2 for vertical and horizontal specimens. The view factor is the fraction of the solid angle subtended by the radiant panels at the specimen face. View factors were calculated [5] for the two orientations with the panels nearly touching along the edges in each of the four sides and a separation of 13, 25, and 38 mm at the corners. The 38-mm opening was chosen for the actual design because the full specimens could be viewed through these openings and because the flux range across the specimen faces did not exceed 10 percent. Various positions on the specimen are indicated as I, II, III, etc. These correspond to center, corner, or edge, as indicated.

The size of the array of radiant panels is 600 x 600 mm (2 x 2 ft) and 300 x 600 mm (1 x 2 ft). For the specimen sizes chosen, this size array of panels is adequate because the specimens are outside the zone of convective heating by the panels.

2.2.4 Measurement of Heat Release Rate

Several modes of measurement of the heat release rate were available. Temperature rise of stack gases due to sample combustion was the earliest method, which is still used in the OSU calorimeter [3]. This constitutes, effectively, a sensible enthalpy measurement. The long response time of such an instrument was much reduced by use of a "substitution" burner. This scheme was to maintain a constant stack gas temperature by reducing the fuel to the substitution burner as combustion of the specimen added heat. Stack temperature is held constant by a temperature controller acting on the fuel flow to the substitution burner. The measurement of the decrease in substitution burner fuel feed directly represents the specimen heat release. Response using both types of readouts in the first Bureau heat release rate calorimeter, NBS I, to two square wave methane calibrations is shown in figure 3. With the isothermal substitution burner technique, full response to a step change was realized in less than 5 seconds. When the instrument was operated in the sensible enthalpy measurement mode, full response took much longer than 1 minute.

Recently, the lag inherent in the sensible enthalpy technique was reduced by the use of a "thermal inertia compensator" with the OSU calorimeter; this is being evaluated by an ASTM task group. A numerical compensation scheme also has been presented by Evans and Breden [6].

Another mode of measurement of the heat release rate is by oxygen consumption. A significant number of materials liberate a nearly constant amount of heat for a given amount of oxygen consumed. While there is no inherent time lag associated with oxygen consumption, there is a lag associated with measurement of oxygen concentration.

² Certain commercial equipment, instruments, or materials are identified in this report in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the material or equipment identified is necessarily the best available for the purpose.

The operating choice between the isothermal method and the oxygen consumption method now appears to be a matter of preference. When the NBS II calorimeter was designed, the isothermal method was obviously the best choice. NBS II can be operated in any of the three modes. Experience and the specific task at hand will probably determine the more desirable mode.

2.2.5 Substitution Burner

A blast burner, forced air and gas, was chosen for a substitution burner. This design permits the burner to be placed in the wall of the calorimeter, out of the stream of combustion products without introduction of burner thermal mass into the stream. The burner chosen was an Eclipse 40 AH² burner designed for heating air. It has infinite turndown, i.e., the ability to combust the fuel gas at rates from maximum to zero. This is a desirable characteristic since the entire capacity of the burner is then available for the measurement of heat release rate.

Control for the substitution burner was provided by a Leeds and Northrup Electromax III² temperature controller with proportional band, reset, approach, and lag controls. The controller operates into a Kepco P114783² power supply which drives the control valve. This control valve is a fast response, solenoid modulating valve of small size: Brooks Instrument Control Valve, 8948B037BMA². The small size of this valve necessitated use of high pressure gas, 100 kPa (15 psi), to get sufficient gas through it to accommodate a 500 kW/m² heat release rate for 300 x 300 mm specimens.

2.2.6 Pilot Ignition of Specimens

Two types of specimen ignition are provided. Electric spark ignition pilots are used at the top of vertical specimens and over the edge of horizontal specimens. Electric spark ignition pilots avoid the difficulty of gas pilots which can be blown out or snuffed out by specimen decomposition products.

A curtain pilot also was provided as an option to engulf the face of vertical specimens. This pilot is stationary within the calorimeter; the specimen is elevated into position beside it. A bottom point pilot, such as that used with the OSU calorimeter, can be operated from the same source. Spark ignition of the pilot flames is provided.

2.2.7 Specimen Carrier - Calibration Burner

Since radiant panels surround the specimens on four sides, specimens had to be introduced from below. A rolling carriage-elevator arrangement was devised for the purpose. In the forward elevated position, the specimen holder is accessible at the front of the calorimeter. This is where the specimen is loaded on a holder, attached to the load cell. The elevator is lowered with a pneumatic cylinder, the carriage pushed back, and the elevator raised to bring the specimen within view of the radiant panels. Figure 7 shows this operation pictorially.

The elevator was made with two sides. The second side is used to carry a diffusion flame pipe burner, which provides a convenient mode for calibration. Were calibration sufficiently infrequent, the line pilot could be used for this purpose and the second position could be used to mount a second specimen holder. If both vertical and horizontal specimen holders were mounted, tedious exchange of holders could be avoided. However, this would require an additional load cell arrangement for measuring the mass loss.

2.2.8 Measurement of Flow Rates

Orifice gauges were used to measure the flow rate of dilution air, air to the substitution burner, air to the radiant panels, and gas to the radiant panels. Mass flow meters were provided to measure gas to the substitution and calibration burners. A variable area flow meter was provided to check the accuracy of the mass flow meter on the calibration burner.

2.2.9 Dilution Air-Cooling Towers

Dilution air, additional air beyond that required for combustion either of the specimens or of gas from the various burners, is introduced for several reasons. In the event that energy balance is to be calculated, sufficient dilution air will permit use of thermal data for air rather than having to use data for the individual combustion products. Another reason is that dilution air lowers the temperature of the instrument significantly.

The possibility of ignition of the premixed gas and air within the radiant panels was thought to be a problem. Irradiation of one panel by another in the box configuration could raise the temperature of the metal parts of the burner sufficiently that the premix would ignite and destroy the porcelain burner panels. To avoid this possibility, dilution air was introduced through an air manifold at the base of the calorimeter and directed up vertical pipes, called cooling towers to the back of each radiant panel. One cooling tower was located behind each vertical burner array. Parts in each tower directed air at the bolts holding each burner onto its manifold. The air was to cool the back of the burners.

The cooling of the burner backs themselves described above was found to be unnecessary. Operation at maximum flux with no dilution air flow produced a temperature of only about 400°C at the back of the porcelain panel plaques. The ignition temperature for natural gas-air mixtures is about 500°C. Even though the cooling of the burner backs was found to be unnecessary, their use was found to be desirable for keeping the sheetmetal shrouds cooler. Cool shrouds help keep the room housing the calorimeter more comfortable to work in and reduce the possibility of personnel getting burned.

2.2.10 Specimen Holders and Load Cell

Vertical and horizontal specimen holders were designed with water-cooling elements between the holder and load cell. The whole frame for the vertical holders was water-cooled. Connection for the cooling water was done through pliant coils of copper tubing which did not rub or touch anything. Friction from touching would result in "backlash" and step-like weight versus time curves. The load cell was mounted in a five-sided stainless steel box, each side of which is water-cooled.

2.2.11 Safety Interlocks

There are explosion hazards associated with the use of gaseous fuels. Safety interlock devices and conservative design were employed to reduce the hazard. The calorimeter has the following features designed for safe operation. The flue gases from the calorimeter go directly through the building roof by way of the instrument's own stack; bypassing the building's central hood and duct exhaust system. This stack assures that uncombusted natural gas does not enter the building's exhaust ducts and create an explosion hazard. There is a pressure interlock switch on the dilution air to the instrument downstream of the butterfly throttling valve. If the dilution air is not flowing to the instrument, the interlock switch

causes the main gas valve to close. This avoids accumulating explosive concentrations of gas within the instrument. The instrument stack and dilution air pressure switch constitute the main safety features. An additional safety device consists of a "flame rod". This is a device which senses whether there is a flame on the surface of the radiant panel and causes the main gas valve to shut down if the flame goes out. This device is installed on the radiant panels that are operated for both vertical and horizontal specimens. The hazard that this device protects against is the gas going off, then coming back on again and not being ignited. With the flame rod, the main gas valve will close when the flame goes out and will not reopen if the gas comes on later.

A hood over the specimen loading and unloading station protects the operator against fumes from the specimen residues. Curtains can be installed if greater air velocity past the operator is needed.

3. CONSTRUCTION

3.1 Mechanical Details

Mechanical details of the calorimeter are described in the group of engineering drawings listed in table 2. An artist's rendition of the instrument, figure 4, illustrates the main features. A dimensioned view of the calorimeter is shown in figure 5. The calibration burner is shown within the array of radiant panels, in the sectional view of figure 6. The cooling towers carrying dilution air are also shown. The main base of the instrument is a rectangular frame of 150 x 300 mm rectangular tubing about 1 x 1-1/2 m (3-1/2 x 4 ft) overall. This frame also serves as the dilution air manifold. An angle iron frame on the main instrument frame supports the shrouds which have windows for viewing the burning specimens. The shrouds constitute a box about 1-1/3 m in length, width, and height. A transition to a rectangular stack sits atop the shroud support frame. The thermocouples which monitor flue gas temperature are about 1-1/3 m above the top of the transition. The whole instrument is supported by rigid angle iron legs welded to the dilution air manifold. The sample carriage-elevator arrangement rolls between the legs on aluminum support rails hung from the dilution air manifold. Figure 7 shows the operation of the carriage-elevator moving the vertical sample holder from outside the calorimeter, where samples are loaded and unloaded, to the inside of the calorimeter exposing them to the radiant panels.

3.2 Gas and Low Pressure Air Distribution

Gas and low pressure air piping to the calorimeter are shown in figure 8. The air pressure is applied by the blower at 5.2 kPa (12 oz/in²).

3.3 High Pressure Air and Water Distribution

A schematic drawing of high pressure air and cooling water to the calorimeter is given in figure 9. The legend for figures 8 and 9 is found in figure 10.

3.4 Electrical

Electrical power for the calorimeter is supplied by a single 120 volt 60-cycle 20 amp circuit. A single switch on the control panel deactivates the calorimeter, electrical, air blower, water, and instrumentation. Compressed air to hold the sample elevator up remains always on.

3.5 Instrumentation

Flow rate of gas to the substitution burner is measured by a Datametrics 800 LM² mass flow meter of 0-2.36 L/s (0-5 ft³/min), 0-10 V output. The output signal goes to a signal conditioner which inverts it and provides a continuously adjustable gain. This adjustable gain allows the output to be rationalized so that a recorder may read directly in kW/m² heat release for a given specimen size.

Flow rate to the calibration burner of the specimen pilots is read on a second identical mass flow meter without a signal conditioner. A variable area flow meter is used optionally in series with the mass flow meter to check the calibration.

Specimen weight is measured with an Automatic Timing Control series 6005C² weigh cell and demodulator model 6101-F². The weigh cell range is 0-2 kg (0-4 lb) with 12 kg (26 lb) tare weight adjustment.

The smoke meter was designed by J.S. Steel. It consists of a laser light source, Metrologic ME-620², 0.8 mW, and a 1P39 phototube receiver with a logarithmic amplifier. The output is 0-5 volts, which corresponds to 0-5 optical density units.

The combustion meter is a Leeds and Northrup total combustible gas analyzer, #7867-4-3-1-005-0-2-1-00-0². The output is 0-10 V for 0-5 percent combustibles.

A Gardon flux gage, 0-100 kW/m², is used to monitor the flux incident on the specimen, either vertical or horizontal. The output is about 0.0125 mV/kW/m² flux.

A six point, console Rikadenko recorder² is used adjacent to the calorimeter. The data can also be taken digitally, stored on discs, and later reduced on a minicomputer in another building.

4. OPERATING PROBLEMS

4.1 Start-Up Problem

Completion of the instrument took many months. The large parts were completed by the main shop in July 1977. Sheet metal, plumbing, and electrical work were done in mid November 1977; instrument-type plumbing, electrical, and mechanical work went on until June 1978.

Once the basic assembly work was completed and start up testing begun many new problems appeared. Even with the commercially purchased ignition system, reliable panel ignition took much trial and error fitting the sample load cell and carriage-elevator worked well when the calorimeter was cold but became erratic or jammed when the calorimeter was at normal operating temperatures. These problems were ultimately overcome by reinforcing the calorimeters base and judicious use of water cooled shielding.

4.2 The Stack Problem

The single most serious problem in operation has been that of fluctuations in rate of flow of gas to the substitution burner, under steady operating conditions. These fluctuations were ultimately found to be the result of variable wind velocity at the top of the stack, resulting in a pressure differential as large as 50 to 75 Pa (0.2 to 0.3 in H₂O).

The best baseline uniformity, i.e., the gas flow rate at steady conditions with no specimen in the calorimeter, corresponded to ± 2 to 3 kW/m^2 on $300 \times 300\text{-mm}$ specimen basis. During gusty winds preceding a rain squall, the baseline varied randomly over a range of 100 to 150 kW/m^2 . On a gusty winter day, baseline variation was often 50 to 100 kW/m^2 . The tolerable maximum is about 10 to 20 kW/m^2 .

The first approach to solving the stack problem was to install (a) a venturi type cap on the stack to prevent "downrafting" and (b) a barometric damper in the stack. The rationale was that the venturi cap would assure a negative stack pressure and the damper would modulate the negative pressure to a constant value. This approach did not solve the problem. The stack pressure was not sufficiently negative for the barometric damper to function.

Two other approaches were tried unsuccessfully:

1. Air flow through the instrument was increased to a maximum so that the stack pressure change due to the wind would be a smaller part of the total pressure drop. The fuel flow variation was reduced, but large positive pressure in the calorimeter, about 25 Pa (0.1 in H_2O), was objectionable. Combustion products leaked out into the room causing eye and throat discomfort, and possible toxicity problems.
2. Air flow was minimized and the substitution burner was operated at capacity. The objective was to maximize the draft in order to cause the barometric damper to function. The draft produced at 430°C was inadequate, about 12.5 Pa (0.05 in H_2O), to operate the damper. (Insulating the stack would also increase the draft. Calculations indicated only about a 10 percent increase in draft could be obtained, which was considered to be inadequate.)

Increased stack height to get above eddies caused by the building did not appear to be a viable solution. Significant pressure variations occurred even in the main stack, which is about 7.6 m (25 ft) taller than the calorimeter stack. Architectural practices at the Bureau militate against a stack taller than the main building stack.

The variable stack pressure problem possibly could be solved by installing a draft inducing blower and an adjustable damper in series in the stack. The objective is to create a large pressure change going up the stack such that the pressure variation due to eddies is relatively small. A pressure of 1.25 kPa (5 in H_2O) appears to be a good goal. The capacity of the blower must be adequate to handle the volume of the calorimeter stack gases. Pressure in the calorimeter itself should be neutral. Calorimeter pressure can be adjusted by varying the damper opening. The barometric damper should be removed to reduce the effect of pressure changes due to opening and closing building doors on calorimeter operation. This design change might improve the stability of the baseline but at present it has not been tried, limiting optimum calorimeter operation to relatively calm days.

5. OPERATION

5.1 Flux Range

The radiant panels operate over a pressure range of about 100 Pa (0.4 in H_2O) to a design maximum of 870 Pa (3.5 in H_2O). The associated flux range is about 25 to 80 kW/m^2 .

5.2 Start-Up Procedure

Check main panel to be sure all switches are OFF.

Turn main power switch on panel to ON (level handle). This will activate the following functions:

- main panel power on,
- opens cooling water solenoid valves,
- energizes both duplex receptacles on power panel,
- energizes control system,
- energizes data system,
- energizes air pump and combustion analyzer, and
- energizes laser on smoke meter.

Turn air blower on by pushing green START button.

This will turn on the air blower in the equipment room -- wait for yellow light -- and will close the air pressure safety switch to supply power to main gas valve.

If yellow light does not come on within 2 minutes, increase the air flow by opening valve 35.

Open gas valve 8 for samples being tested in the vertical sample holder or valves 7 and 8 for samples being tested in the horizontal sample holder.

Open gate valve 32 for vertical samples or valves 32 and 33 for horizontal samples to 250 Pa (1 in H₂O) or more of premixed gas and air.

Turn radiant panel switch on main panel from OFF to IGN and hold on IGN until red light comes on.

This will activate the following:

- spark igniters on face of radiant panels will ignite panels, and
- main gas valve will be held open by flame rod relay when flame is established and pilot light comes on.

Turn substitution burner switch from OFF to IGN to ignite pilot flame for substitution burner.

The following controls are located on the control panel. Turn the substitution burner controller toggle switch from MANUAL to AUTO.

Turn voltage control knob to the maximum setting that does not open substitution burner valve, about "11 o'clock".

Turn temperature controller knob to 220 C or greater.

Turn toggle switch to signal conditioner to ON.

Turn toggle switch for power to controller ON to activate the substitution burner.

Turn combustion analyzer toggle switch to ON.

Turn smoke meter analyzer toggle switch ON.

Turn toggle switch on outlet box on main panel ON to activate laser, smoke pump, and smoke analyzer.

Turn analog to digital converter toggle switch ON.

Turn both modems table units ON by operating rocker switches on back of units.

Turn on strip chart recorder by switching ON main power switch on front of recorder.

Turn on all rocker switches for each individual pen, on top of recorder.

Set chart speed as desired.

This concludes the start-up procedure.

5.3 Operating Procedures

Once the calorimeter is started, it is necessary to set several variables to the desired levels. First, it is necessary to establish the orientation of the sample, either horizontally or vertically. Second, the size of sample to be tested must be established. The standard maximum size for horizontal samples is 150 x 300 mm (1/2 x 1 ft) and for vertical samples is 300 x 300 mm (1 x 1 ft); smaller sizes may be used. The thickness of the sample is limited primarily by the sample holder being used. Third, the type of sample ignition must be established -- either gas flame, electric spark, or no igniter. Once these variables have been established, NBS II must be adjusted to meet them. Horizontal samples require a pan or tray approximately the same size as the sample to be tested to rest on the insulation above the load cell.

Vertical samples require installation of a special frame to be mounted on the load cell. This can be accomplished by moving the carriage elevator so the load cell is outside the calorimeter. This is the normal position of the carriage-elevator when loading and unloading samples. The vertical sample holder is then bolted to the load cell, and the cooling water connections are made. The flux meter should be attached to the side of the sample holder and the water and electric lines for it connected. The water lines must be pliant and must not touch each other or any part of the frame or the load cell output will be erratic. Once the vertical sample holder is in place, samples are inserted into the holder backed up with ceramic insulation board and secured with spring clamps.

With the sample holder positioned, it is necessary to install the sample igniter system, if one is going to be used. The spark igniter on the horizontal sample holder involves the use of a spark plug with a long tip and ground electrode. This spark plug is held on a metal strap which can position the electrodes over or near the sample. Voltage for the spark is picked up by way of an 18 cm (7 in) long wire which engages a high voltage wire frame inside the stationary part of the calorimeter. This igniter can be attached and adjusted while the calorimeter is in operation.

The spark igniter for the vertical sample holder attaches to the top of the sample holder, and employs a similar type of spark plug as the one used on the horizontal sample holder. This spark plug also engages a high voltage wire frame inside the calorimeter. It can be attached and adjusted while the calorimeter is in operation.

The gas igniter for both the horizontal and vertical sample holders uses a stainless steel tube with holes in the end to generate an igniter flame. The pilot flame for the horizontal sample holder may be positioned above or alongside the sample simply by bending the tubing slightly. The igniter flame for the vertical sample is placed in front of and below the sample holder. In this position, either a small igniter flame or a sheet igniter flame may be used. The sample igniter is supplied by way of a solenoid controlled gas valve. To install the gas igniters for either the horizontal or vertical holder, the calorimeter is turned off and allowed to cool. The back panel is removed by taking out fourteen 10-32 hex head screws and lifting it off. This will give access to the fittings on the solenoid controlled gas line. The appropriate igniter is connected to the gas line using the compression fittings supplied. The back cover is replaced and the calorimeter started up.

It is now necessary to set the flux levels. With the calorimeter operating, the appropriate sample holder is inserted into the calorimeter so that the flux meter is facing the radiant panels. Note that when using the vertical sample holder, only those radiant panels facing the sample will be used. The other half of the radiant panels will be turned off. Adjust the gate valves 7 and 8, or valve 8 only for vertical samples, to obtain the desired flux. An hour warm-up is required to assure that a steady state has been reached.

The substitution burner is set to about 1.9 L/s (4 SCFM) on natural gas, as read on the mass flow meter*. This will yield near maximum turn down measuring capacity without seriously affecting the elevator operating transient. Settings of below 1.9 L/s (4 SCFM) or above 2.1 L/s (4.5 SCFM) tend to give an excessively slow recovery time when the elevator is operated. Some samples may cause the substitution burner to shut down completely; hence, peak heat release rate from a sample will not be ascertained. If this occurs, the sample size must be reduced so that peak heat release will be within the range of response of the substitution burner.

In setting the dilution air, it is desirable to control the dilution air going into the calorimeter to provide a neutral pressure condition near the specimen height level in the calorimeter. To accomplish this, adjust dilution air valve 35 until the manometer reading air pressure in the calorimeter is about zero. Take note that closing valve 35 too far may cause the pressure safety switch to trip out and shut down the calorimeter. If this occurs, open valve 35 back up and restart the calorimeter. It is advisable to strive for a neutral or balanced air pressure, as a strong positive pressure will cause much loss of heated air when the elevator is operated. This heat loss will exaggerate the recovery time for the substitution burner. Likewise, an excessive negative pressure inside the calorimeter will draw in cold air when the elevator is operated, also resulting in a worsening of the recovery time for the substitution burner.

Once the dilution air has been set, it may be necessary to reset the substitution burner gas flow rate back to 1.9 L/s (4 CFM). With the dilution air, substitution burner, and flux set, the calibration procedure should be carried out before testing any samples. Once this calibration procedure is completed, the operation of the calorimeter in testing a sample

* This adjustment is made by setting an appropriate value for the stack thermocouple grid temperature, typically about 220°C.

involves simply inserting the sample in the appropriate sample holder, operating the elevator to bring the sample inside the calorimeter, and operating the switch "ignition specimen pilot" if a spark igniter is to be used. With the gas flame igniter, the "ignition specimen pilot" switch need not be operated as the flame size has been set and is left burning all the time. Once the test is completed, remove the old sample and replace with a new sample and proceed as above.

5.4 Calibration Procedure

Certain elements of the calorimeter should be checked before each day's run. These items include the mass flow meters, strip chart recorder, flow to calibration burner, substitution burner turn down, and the weigh cell. All calibration tests should be performed after the calorimeter has had a warm-up period of at least one hour.

Calibrate the mass flow meters first. To do this, proceed as follows:

1. Turn mass flow meters OFF; set the mechanical zero on the meter face. Needle should read exactly zero with the unit off. If not, adjust the mechanical zero with a small screwdriver.
2. Turn the mass flow meter ON, with calorimeter at operating temperature; proceed to adjust the meter.
3. Adjust zero flow readings.
 - (a) Shut off gas valves 14 and 20 completely, stopping flow to meter sensors.
 - (b) Set function switch to clean and hold for 5 seconds.
 - (c) Set function switch to CALIBRATE and the three-position toggle switch to BAL; the needle should read exactly "0" on the top scale. If not, adjust the BAL screwdriver potentiometer until the reading is zero.
 - (d) Set the three-position toggle switch to its downward position FS. The meter should read full-scale within ± 1.2 cm ($\pm 1/2$ in). Adjust the FS screwdriver potentiometer until the reading is full-scale.
 - (e) Return the three-position toggle switch to its center position, ZERO. The meter should read zero within 1.6 mm (1/16 in). Adjust the ZERO screwdriver potentiometer until the reading is zero.
 - (f) Reopen valves 14 and 20 and let the substitution burner equilibrate.

This completes the electronic calibration of the mass flow meters. The mass flow meters must also be calibrated against the variable area flow meter. This test insures that the mass flow meters have not lost their calibration. It also assures that the composition of the natural gas being supplied to the calorimeter has not changed. To perform this cross check test, turn substitution burner control to MANUAL, open valves 25, 51, 54, and 21; close valves 20, 22, and 53. The gas is now set to flow through the variable area flow meter and the sensing unit, numbers 16 and 24 of the mass flow meters. Make certain that the calibration

burner is properly positioned inside the calorimeter. Open modulating solenoid valve 15 and set control valve voltage knob to approximately "12 o'clock". Open gas cock number 14 and ignite the gas flowing to the calibration burner. Both mass flow meters should read 0.5 L/s (1.0 SCFM). If not, adjust the control valve voltage knob until both meters read exactly 0.5 L/s (1.0 SCFM). Check the variable area flow meter; it should read 0.36 L/s (46 SCFH). If variable area flow meter reads ± 1.0 L/min (± 2 SCFH) different from 0.36 L/s (46 SCFH), recalibrate the mass flow meters. If variable area flow meter reads ± 1.0 L/min (± 2 SCFH) of 0.36 L/s (46 SCFH) (but not exactly), record this difference, as it will be used as a calibration correction. With this completed, reset all appropriate valves to their normal operating position. This completes meter calibration. Just before running any series of heat release experiments, it is necessary to make one final calibration. With all the above meter checks completed and the calorimeter operating, place the calibration burner into the calorimeter, open valves 20, 21, and 25, and adjust the natural gas flow on the variable area flow meter to 0.36 L/s (46 SCFH) using valve 54. Ignite the gas and note the response the substitution burner gives on the strip chart. From this calibration, all subsequent heat release calculations can be made.

The strip chart recorder must be calibrated, as the output from this recorder will be used to set other parameters. First, set the zero set point for all the pens. With all pens operating and the chart running, turn all six attenuator knobs to off (short). All the pens will give a straight line trace at some point on the chart. These pens may be moved to any convenient point on the chart by use of the zero adjuster knobs. With the zero set point established start turning the attenuator on; first pen numbers 1 and 2, the substitution burner, and substitution burner with signal averager to the 10-volt mark (full-scale). These pens should be in the left quarter of the chart when the substitution burner is consuming approximately 2.0 L/s (4.0 CFM) of natural gas. These pens should move toward the right side of the chart as the substitution burner gas flow is reduced. A quick check on this is to simply switch the power toggle switch to the substitution burner controller off and determine if the pens stay within the chart boundaries.

The flux pen (number 3) is turned from zero (short) to 10 millivolts (full-scale). This pen moves from left to right as flux increases. For every 1 millivolt increase in output, the actual flux increases approximately 12.5 kW/m^2 . To check the actual flux, insert the appropriate sample holder (horizontal or vertical) and flux gage and read the output in millivolts from the zero (short) point. Multiply the millivolt output by 1.26, the constant for the flux gages; this will yield the actual flux falling on the flux meter. To increase or decrease flux, use valves 7 and 8.

The optical density smoke meter (pen 4) is turned from the zero (short) set point to the 5-volt full-scale position. This pen moves from left to right and, for every volt increase in output, the observation is increased 10-fold. There is no calibration check for the optical density output.

The combustion meter (pen 5) is turned from the zero (short) set point to the 1-volt full-scale position; the pen will move off the zero point and establish a new equilibrium point. This pens moves from left to right. To calibrate the combustion meter, insert the calibration burner into the calorimeter, and open the solenoid gas valve to the calibration burner by turning the switch "Calibration Burner" to ON. Set the variable area flow meter to 0.36 L/s (46 SCFH). Do not ignite the gas. The pen will move to the right to about 0.65 volts. The pen should register a 0.0712-volt change for every 5.0 L/min (10-SCFM) change in the rotometer. If this ratio changes, investigate the moisture trap and paper filter on the combustion meter sampling line for possible obstruction.

The weigh cell (pen 6) is turned from the zero (short) set position to the 1-volt level full-scale for samples weighing up to 500 grams. Heavier samples may be weighed by turning the demodulator range switch from zero to 100; at this setting, samples of 1000 grams may now be used. The weigh cell is calibrated by simply placing a known weight on the sample holder and noting the response pen 6 yields. It is often quite helpful to start by placing 50 grams on the sample holder and working up to a total of 500 grams. The output of pen 6 should be in step fashion corresponding to the added weights applied. From this calibration, a gram/division ratio may be developed.

6. ILLUSTRATIVE DATA

The first test in the NBS II calorimeter was on a 75 x 30 x 13 mm (3 x 1 x 1/2 in) polystyrene specimen in the horizontal orientation using the isothermal mode of measurement. A tracing of the original heat release rate and mass remaining curves are shown in figure 11.

Heat release rate measurements were made by Babrauskas [7] on black polymethylmethacrylate using the oxygen consumption technique. The samples were 100 x 100 x 13 mm (4 x 4 x 1/2 in) and were mounted horizontally. The mean values for six sets of measurements together with the standard deviation is plotted in figure 12. An extensive series of heat release rate tests were run on wood products by Chamberlain [8]. A tracing of an original data curve for a vertical specimen of ceiling tile, 300 x 300 x 13 mm (12 x 12 x 1/2 in), exposed at 50 kW/m² and measured in the isothermal mode is seen in figure 13.

7. PROGRAM

The new heat release rate calorimeter, NBS II, appears to be a good quality instrument with broad potential for fire research.

The following research program is suggested for its use:

a. measure for representative materials:

heat release rate		specimen size
mass loss rate	vs.	specimen orientation
smoke		air velocity
uncombusted fuel		type of piloting

b. compare the heat release rate measured directly by oxygen consumption in the NBS II;

c. compare the data obtained with NBS II with the data from NBS I, OSU, and instruments based on oxygen consumption;

d. recommend conditions of measurement for heat release rate on the basis of the experience and understanding gained above;

e. publish heat release data for an extended group of materials measured in the recommended way (these data could constitute the beginning of a handbook of data describing combustibility of materials); and,

- f. determine the effect of material composition (carbon, hydrogen, halogens, oxygen) on heat release rate -- compare the heat release rates of cellulosic and synthetic polymers.

8. ACKNOWLEDGMENTS

The development of the new calorimeter was supported in large part by the Society of the Plastics Industry who sponsored Dr. Tordella as a Research Associate at the Bureau from February 1, 1976 through July 31, 1978.

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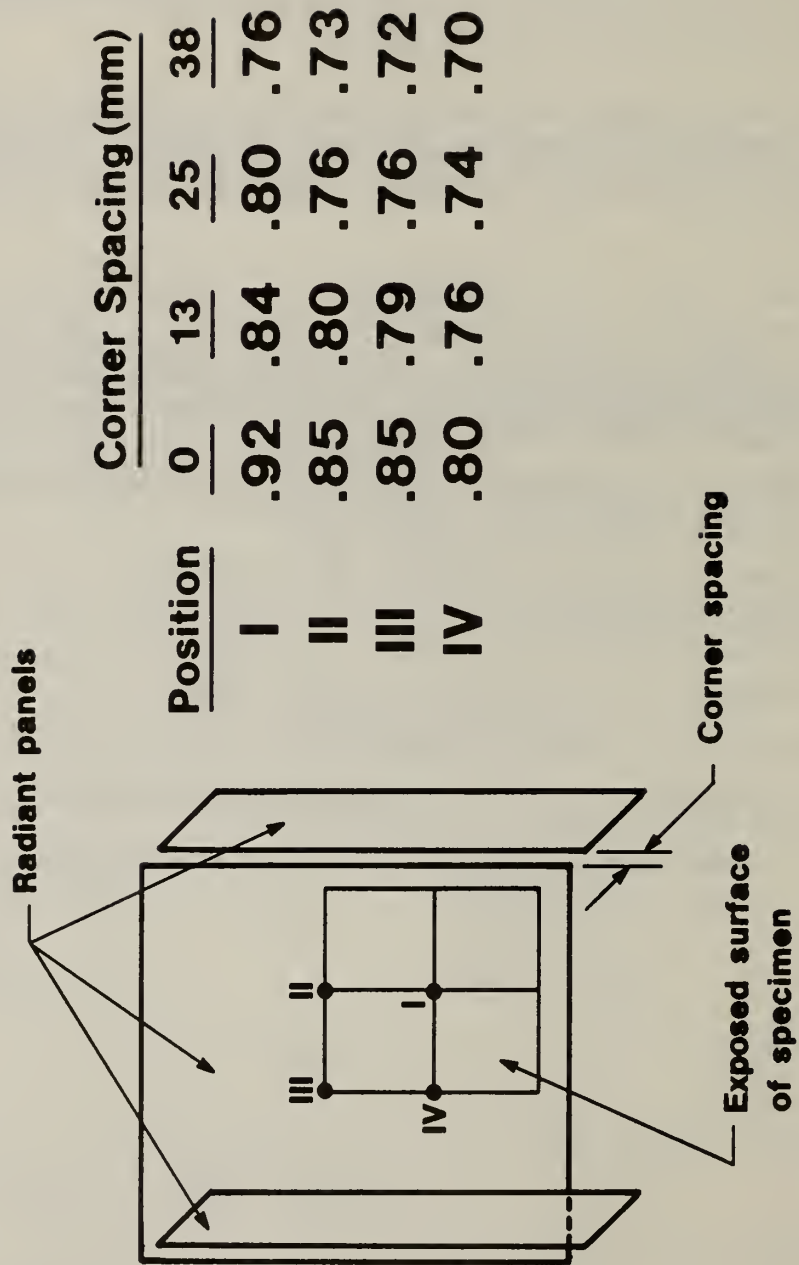


Figure 1. View Factors, Vertical Specimen

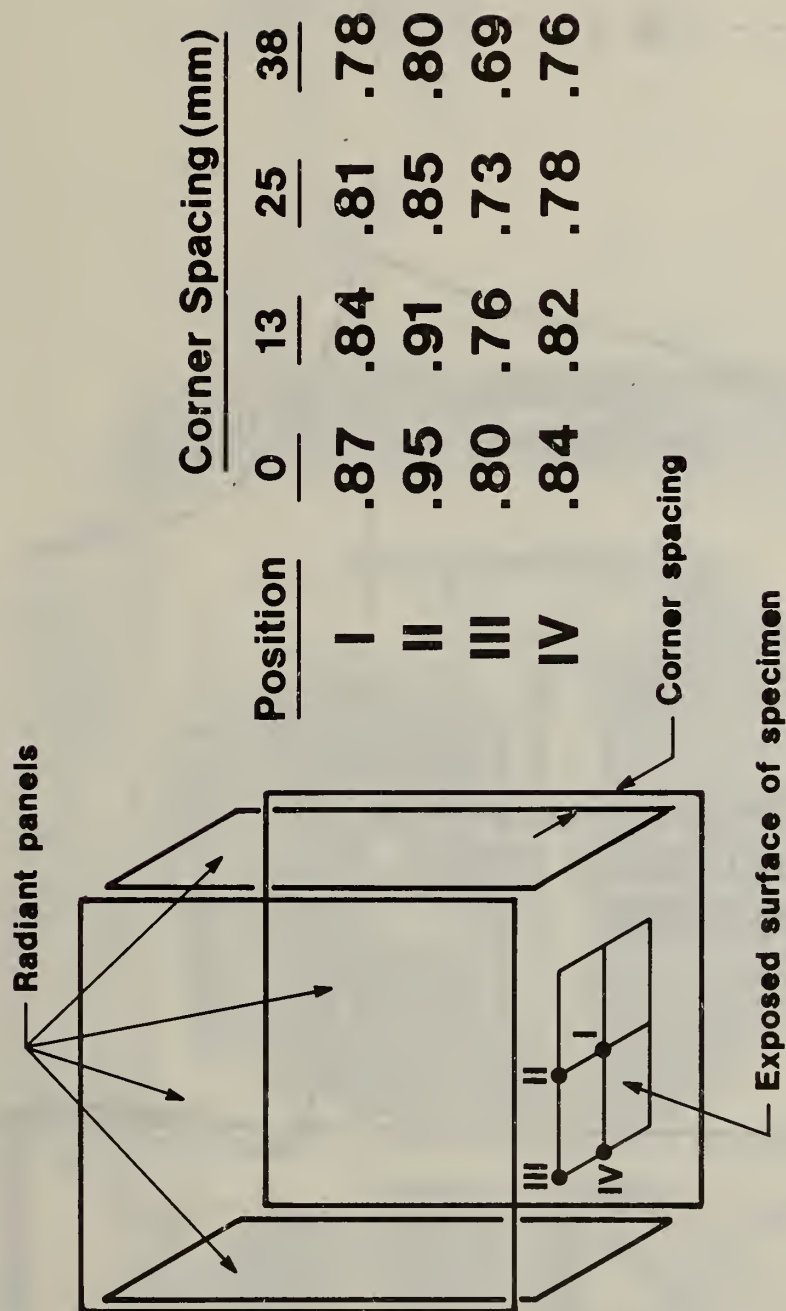


Figure 2. View Factors, Horizontal Specimen

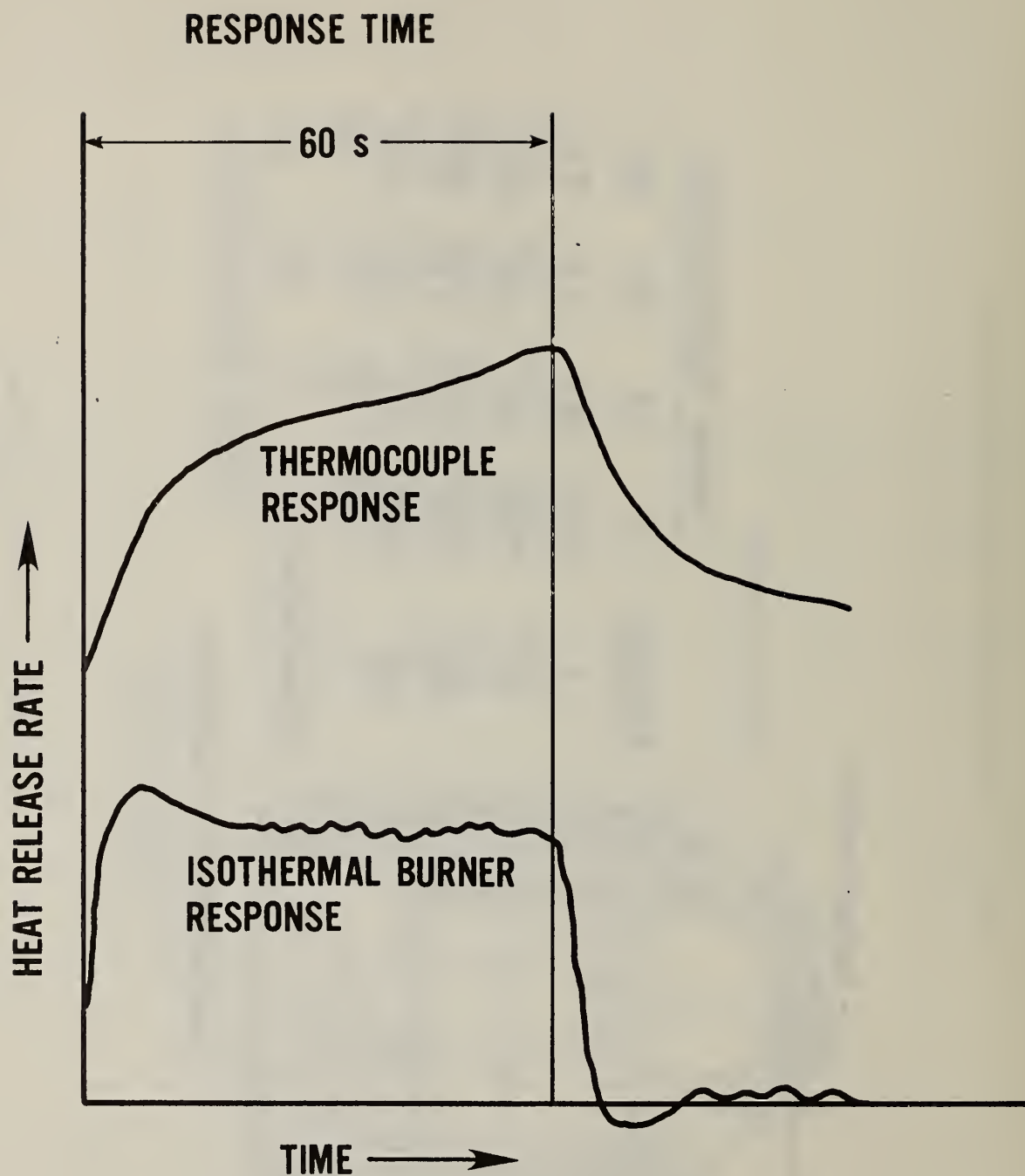


Figure 3. Response Time, NBS I Calorimeter

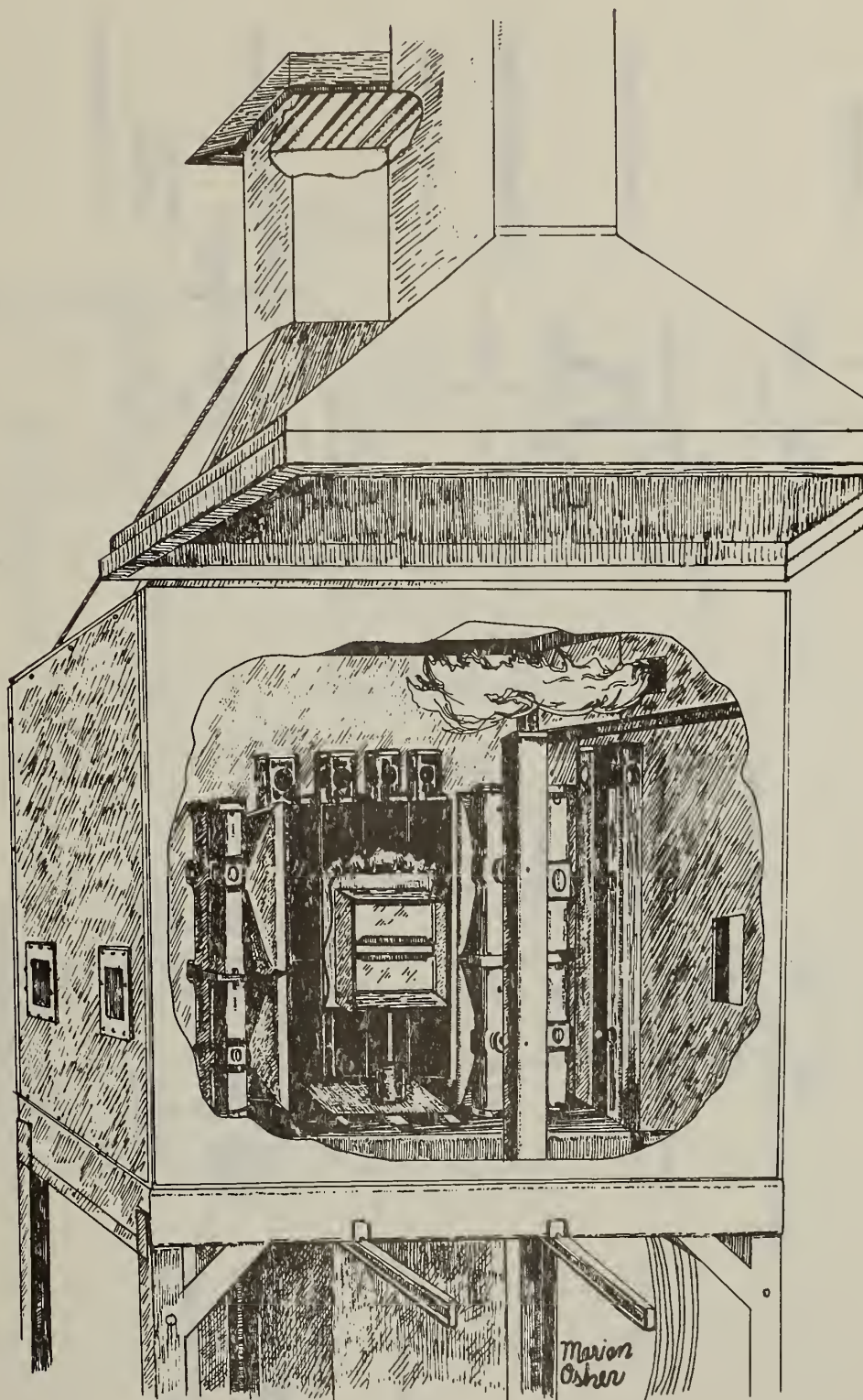
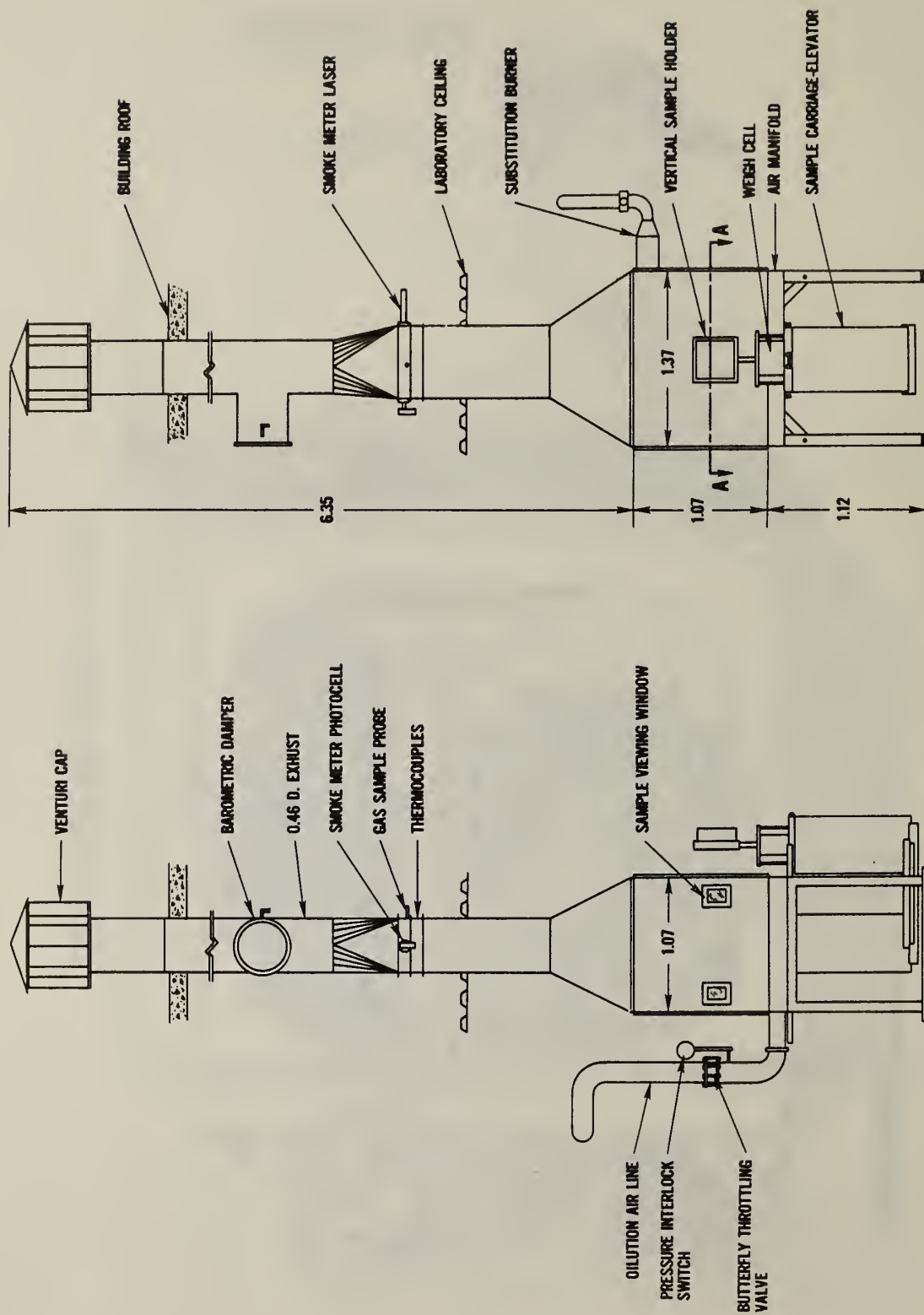


Figure 4. Artist's Rendition, NBS II Calorimeter



ALL DIMENSIONS IN METERS

Figure 5. Dimensioned View of Calorimeter
See Figure 6 for Section A-A

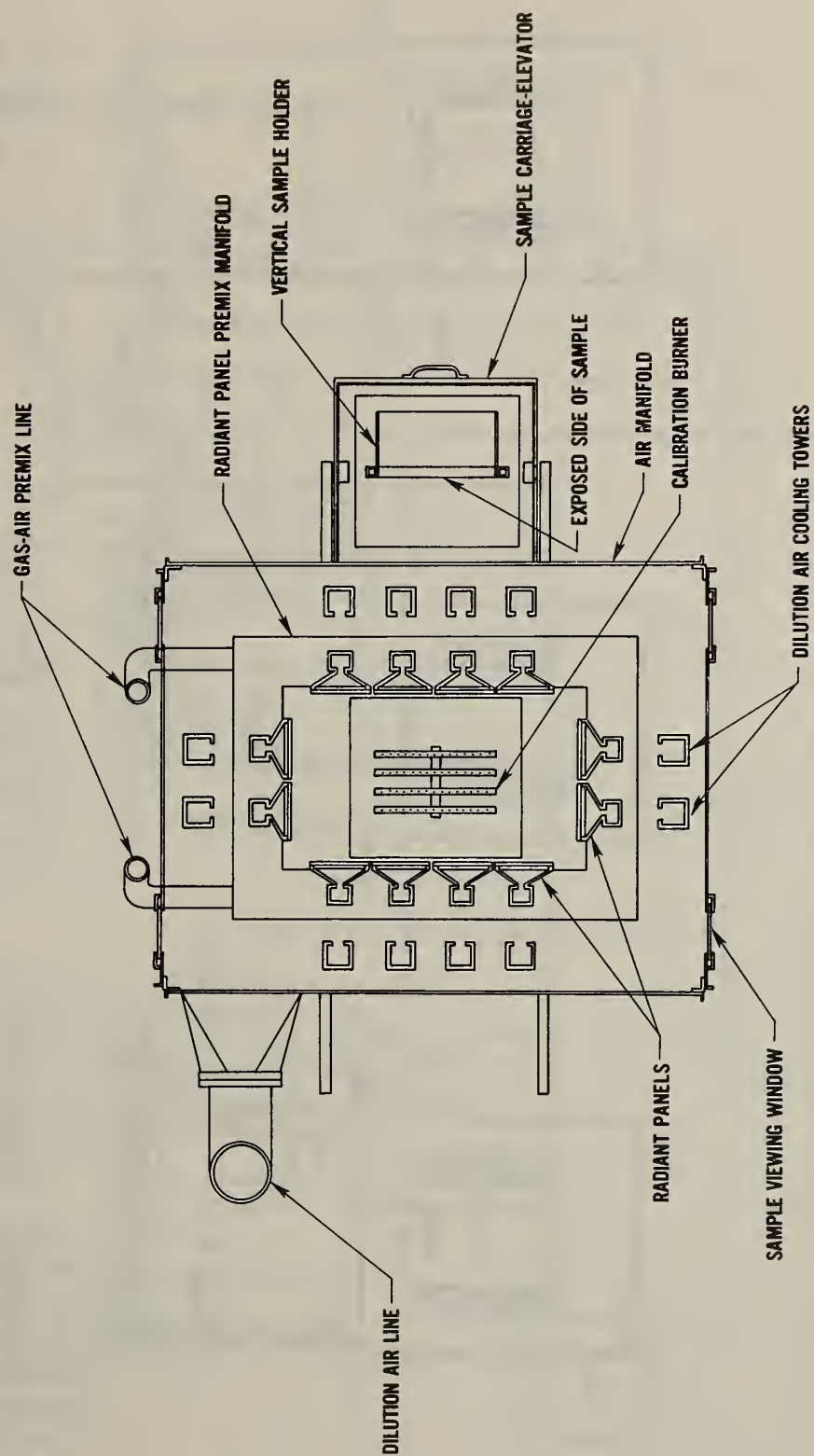


Figure 6. Sectional View A-A of Calorimeter

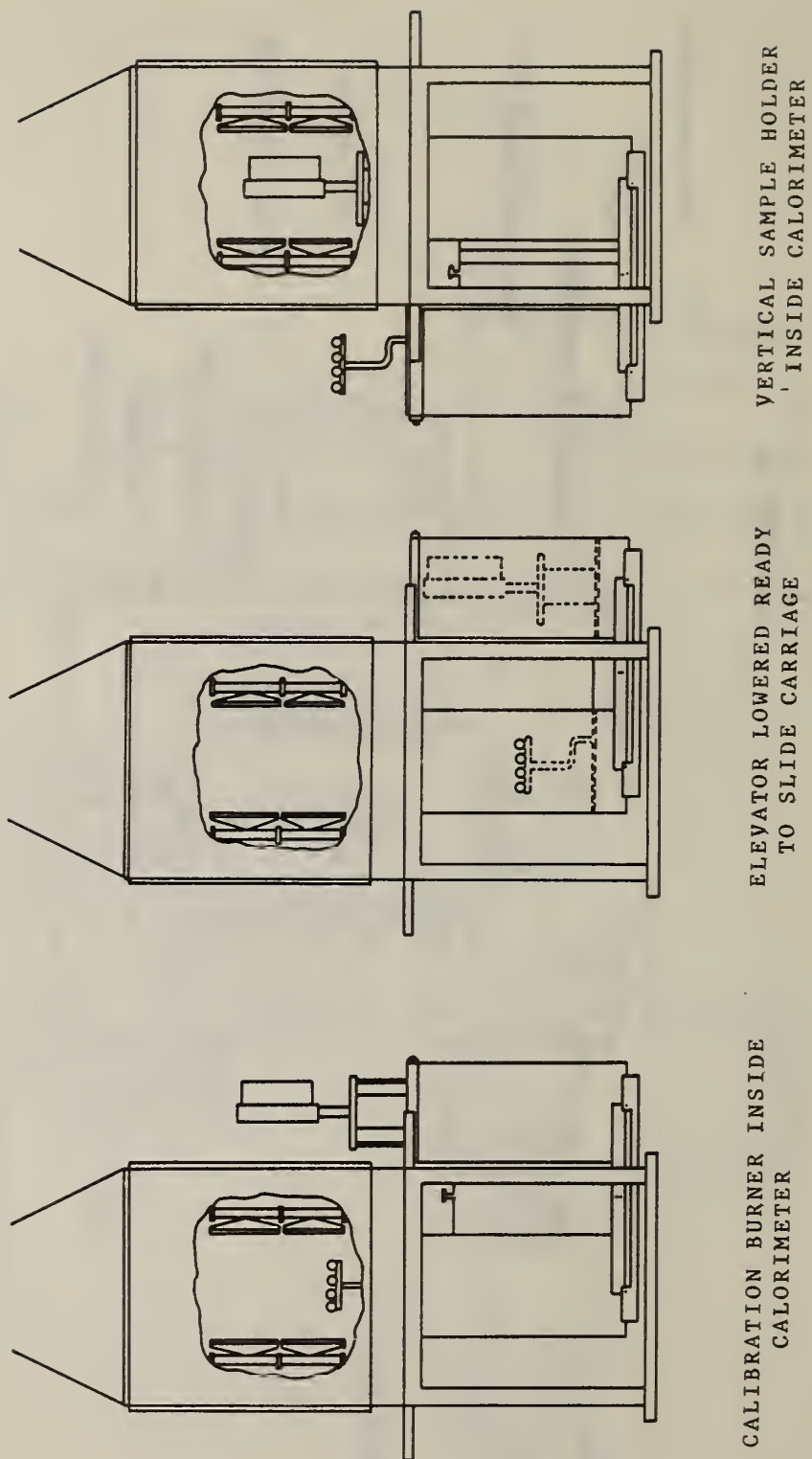


Figure 7. Operation of Carriage-Elevator

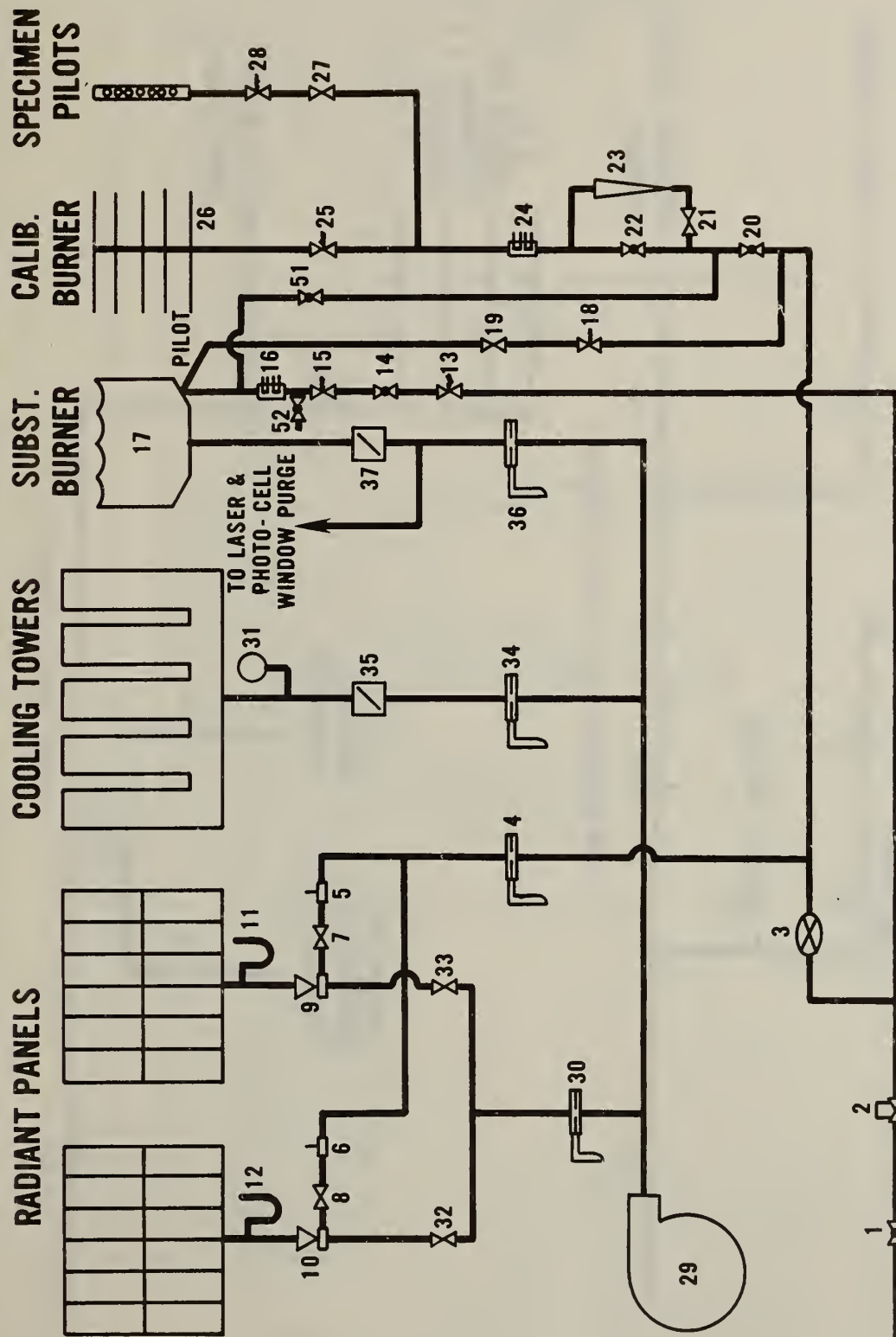


Figure 8. Combustion Gas and Low Pressure Air Schematic

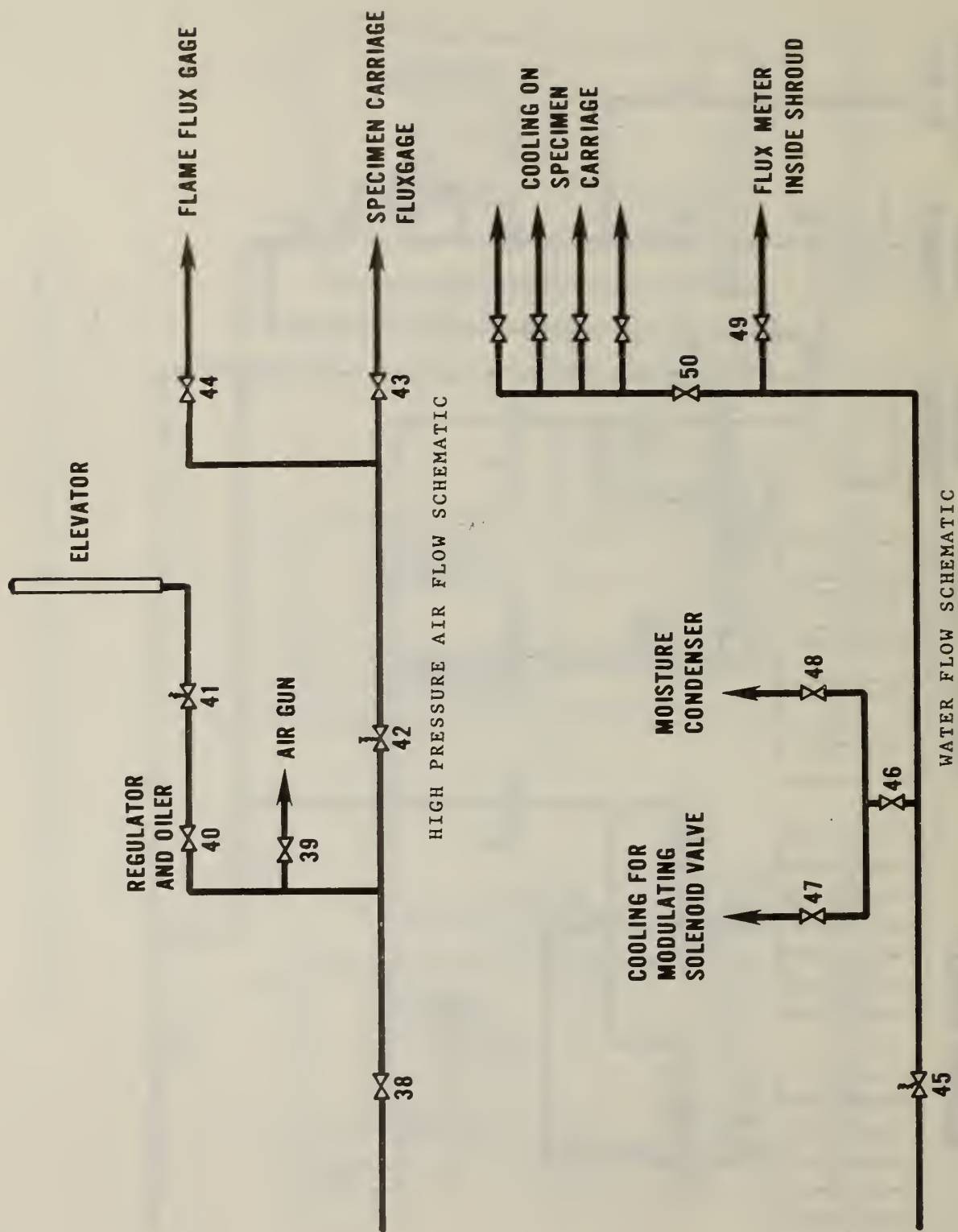


Figure 9. High Pressure Air and Water Schematic

- 1 1" gas cock
- 2 1" motorized gas valve, Eclipse 304-MV-IS-3
- 3 1 1/2" pressure reducer, Eclipse RV-81
- 4 Orifice meter, Eclipse 1" pipe size
- 5 Zero reducer, Eclipse 306 ZG
- 6 Zero reducer, Eclipse 306 ZG
- 7 1" gas cock, Eclipse
- 8 1" gas cock, Eclipse
- 9 Variset mixer, Eclipse 646
- 10 Variset mixer, Eclipse 646
- 11 Manometer, Dwyer 422-5
- 12 Manometer, Dwyer 422-5
- 13 Solenoid valve
- 14 1/2" gas cock
- 15 Modulating solenoid valve, Brooks 948037 BMA
- 16 Datametrics mass flow meter 80 LM
- 17 Air heat burner, Eclipse 40 RAH
- 18 Solenoid valve
- 19 1/4" gas cock
- 20 1/2" gas cock
- 21 1/2" gas cock
- 22 1/2" gas cock
- 23 Variable area flow meter, Dwyer RM2-102-88V
- 24 Datametrics mass flow meter 800 LM
- 25 Solenoid valve
- 26 Calibration burner
- 27 1/2" gas cock
- 28 Solenoid valve
- 29 Air blower, Eclipse SM-8817-7-1/2-E
- 30 Orifice meter, Eclipse 3" size
- 31 Low gas pressure switch, Eclipse C437-H
- 32 1 1/2" gate valve
- 33 1 1/2" gate valve
- 34 Orifice meter, Eclipse 6" pipe
- 35 Butterfly valve, Eclipse 24 BV
- 36 Orifice meter, Eclipse 3" size
- 37 Butterfly valve, Eclipse 112 BV
- 38 1" gate valve
- 39 Air gun valve
- 40 Air pressure regulator and oiler
- 41 Solenoid spool valve
- 42 Solenoid valve
- 43 1/4" needle valve
- 44 1/4" needle valve
- 45 Solenoid valve
- 46 1/2" ball valve
- 47 1/4" needle valve
- 48 1/4" needle valve
- 49 1/8" needle valve
- 50 3/8" ball valve
- 51 1/2" gas cock
- 52 1/2" gas cock
- 53 1/2" gas cock
- 54 1/2" gas cock

Figure 10. Legend for Figures 8 and 9

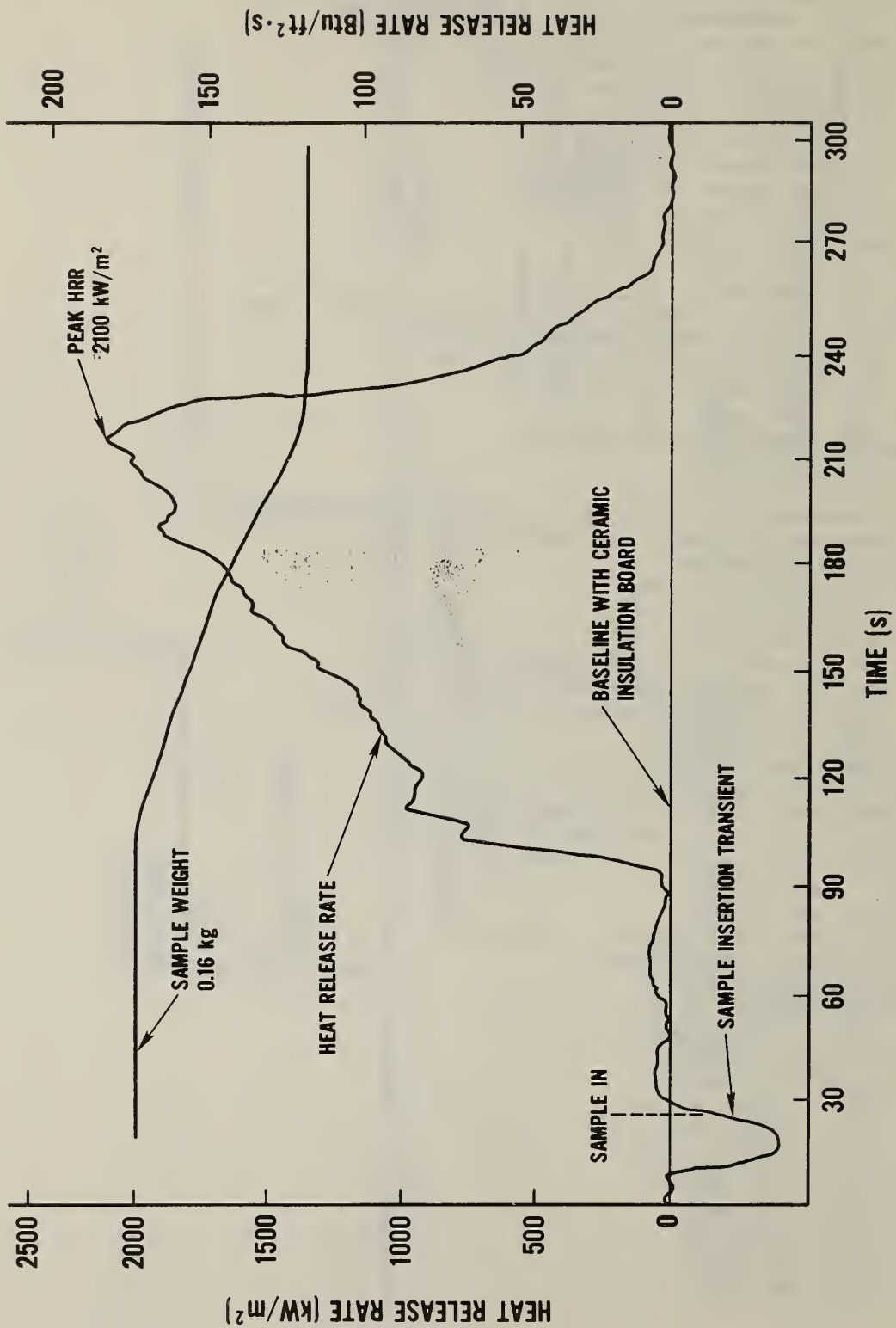


Figure 11. Heat Release Rate and Remaining Mass Curve for Polystyrene at 50kW/m^2 Exposure

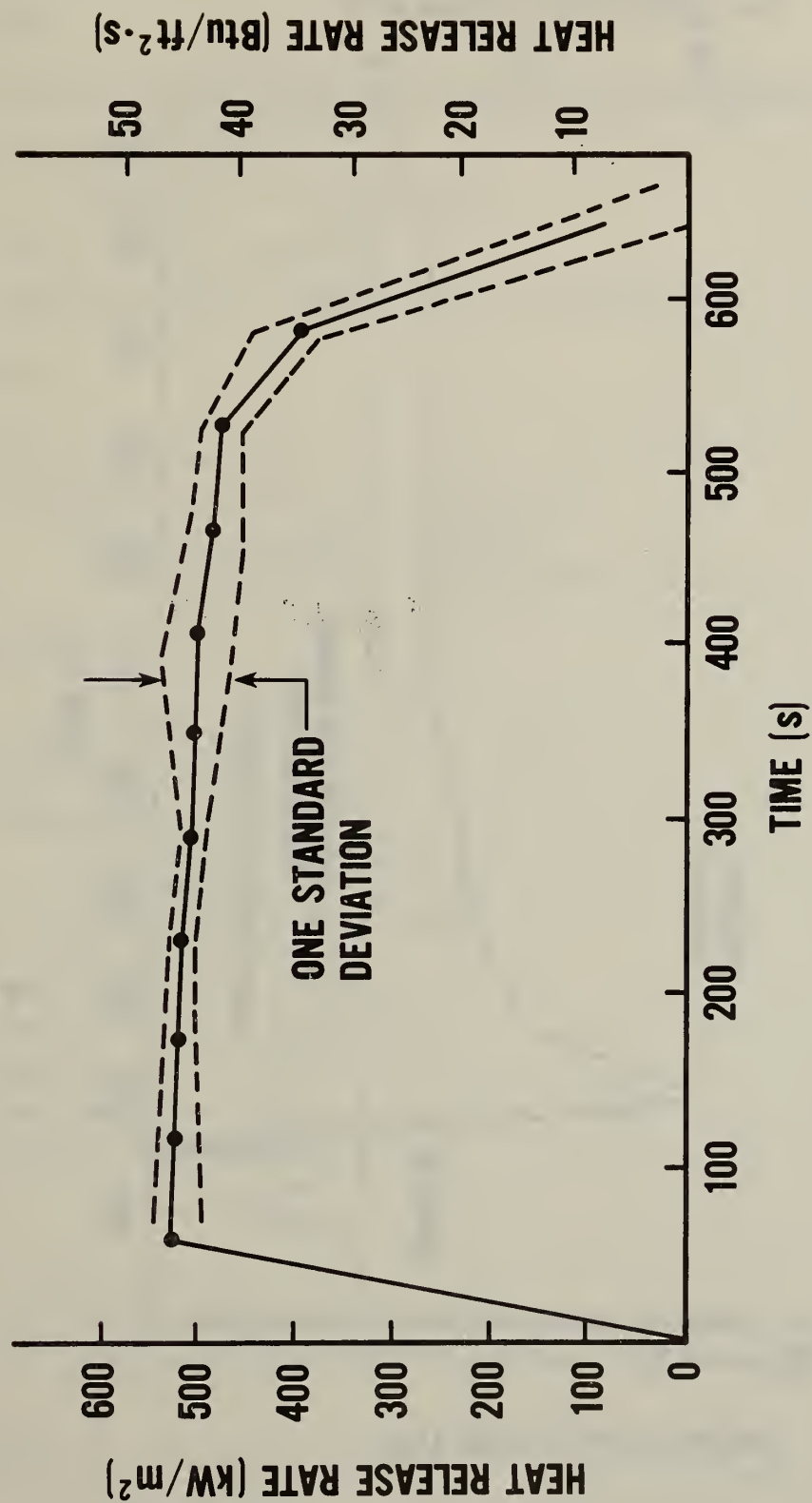


Figure 12. Heat Release Rate of Black Polymethylmethacrylate

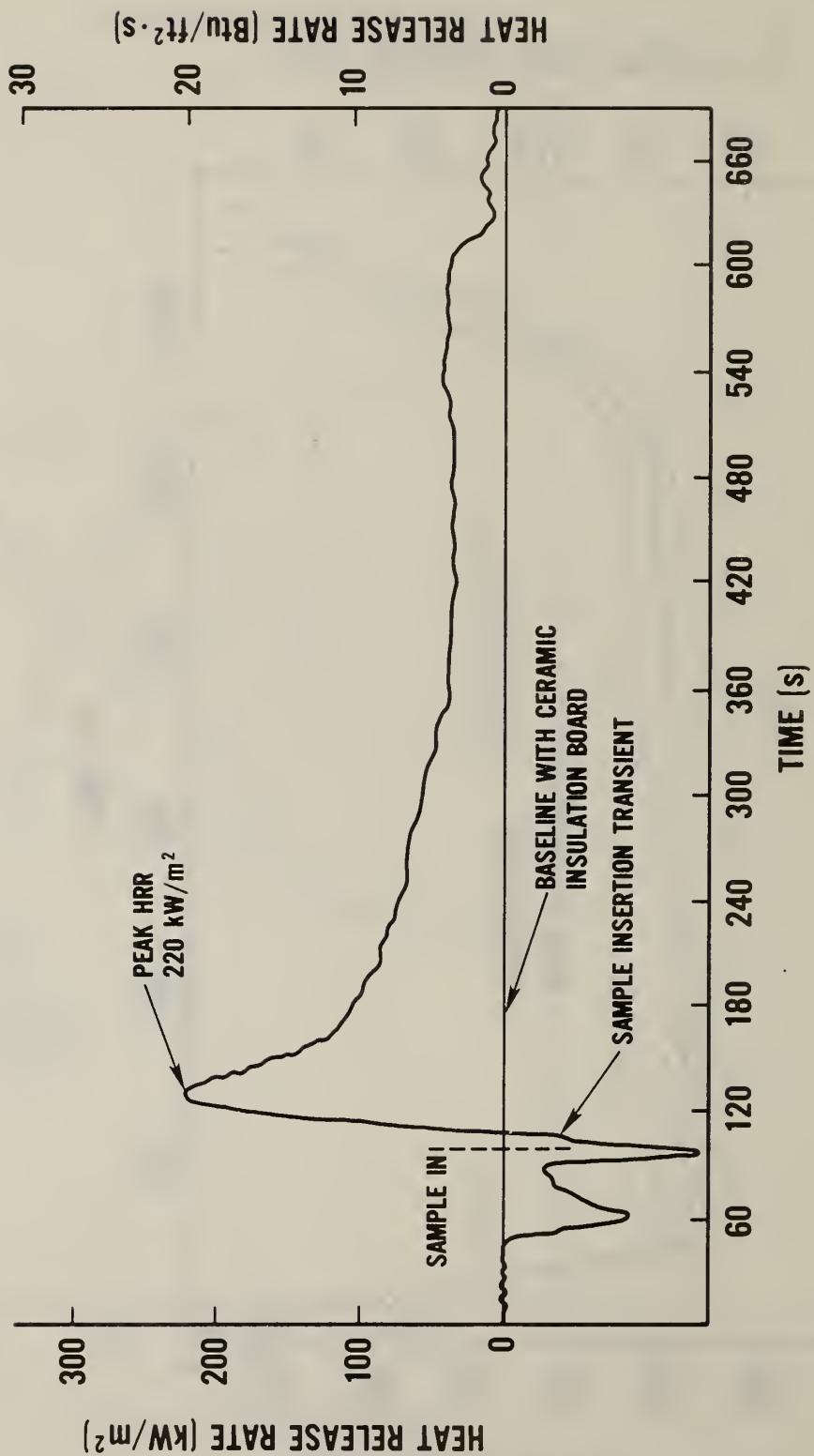


Figure 13. Heat Release Rate of Wood Fiber Ceiling Tile. 50 kW/m² Exposure

Table 1. Specification for New NBS Heat Release Rate Calorimeter

1. SPECIMEN:

external heat flux uniform over specimen surface to 10 percent orientation and size

vertical -- 300 x 300 mm

horizontal -- 150 x 300 mm

view -- full, continuous

ignition pilots -- upper, lower, and curtain

2. CAPACITY:

flux 25 to 80 kW/m²

maximum heat release rate capability

500 to 1000 kW/m² -- vertical

1000 to 2000 kW/m² -- horizontal

3. PERFORMANCE:

warm-up -- 2 hours

stability -- long term

convenient operation

easy specimen placement

flux meter built in

calibration burner built in

low maintenance

accuracy -- $\pm$ 10 percent

precision -- $\pm$ 5 percent

sensitivity -- 5 kW/m²

response speed -- < 5 sec to step change

all flows measurable

4. DATA:

(a) sample heat release and weight loss rates

(b) stack gas/smoke density and heat of combustion

5. RECORDING:

chart recorder and computer terminal

6. MECHANICALLY AND STRUCTURALLY SOUND; SAFE

Table 2. Engineering Drawing Schedule for Calorimeter*

Air Manifold (101087)	Lift Plate (101055)
Air Tower (101067)	Lower Cylinder Br'k't (101053)
Air Transition (101093)	Lower End Frame (101034)
Baffle Support Bar (101046)	Lower Slide Frame (101036)
Base Cover (101077)	Mixer Coupling (101042)
Bearing Carrier (101038)	Pilot Bracket (101064)
Bearing Retainer (101037)	Pipe Adapter (101056)
Burner Bracket (101049)	S.D. Mount Plate (101058)
Calibration Burner (101073)	Short End - Frame (101041)
Curtain Pilot (101086)	Shroud Cover (101084)
Cylinder Top Br'k't (101050)	Side Frame (101040)
Detector Holder (101059)	Small Side Panel (101072)
Detent Block (101063)	Smoke Detector Box (101080)
Elevator Cable Bracket (101045)	Thermocouple Carrier (101074)
Elevator Guide Plate (101065)	Mixer Coupling (101042)
Elevator Lift Bar (101054)	Pilot Bracket (101064)
Elevator Plate (101082)	Pipe Adapter (101056)
Elevator Shroud (101066)	S.D. Mount Plate (101058)
Fluxmeter Bracket (101044)	Short End - Frame (101084)
Frame (101092)	Side Frame (101040)
Gas/Air Manifold (101091)	Small Side Panel (101072)
Guide Rails (101083)	Smoke Detector Box (101081)
Heater Banks (101090)	Thermocouple Carrier (101074)
Heat Chamber Frame (101068)	Thermocouple Carrier Front (101076)
Holder Backing (101079)	Thermocouple Chamber (101075)
Holder Slide (101080)	Tie Block (101052)
Horizontal Specimen Holder (101085)	Transition - Large Side (101069)
Insert, Weigh Cell Insulator (101047)	Transition - Short Side (101070)
Insulator, Weigh Cell (101048)	Vertical Carrier Guide (101035)
Large Side Panel (101071)	Vertical Sample Frame, Water-Cooled (101088)
Laser Insulator (101057)	Weigh Cell Shroud (101089)
Laser Mount (101061)	Weigh Cell Shroud Car (101078)
Laser Plate (101060)	Weigh Cell Stand-Off (101051)
Latch Pin Holder (101062)	

* Copies of the engineering drawings for the calorimeter may be obtained from the Engineering Design Office of the National Bureau of Standards.

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET (See instructions)	1. PUBLICATION OR REPORT NO. NBSIR 83-2708	2. Performing Organ. Report No.	3. Publication Date June 1983
4. TITLE AND SUBTITLE Development of a Calorimeter for Simultaneously Measuring Heat Release and Mass Loss Rates			
5. AUTHOR(S) John Tordella and William H. Twilley			
6. PERFORMING ORGANIZATION (If joint or other than NBS, see instructions) NATIONAL BUREAU OF STANDARDS DEPARTMENT OF COMMERCE WASHINGTON, D.C. 20234			7. Contract/Grant No. 8. Type of Report & Period Covered
9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (Street, City, State, ZIP)			
10. SUPPLEMENTARY NOTES ☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.			
11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here) A heat release rate calorimeter (designated as NBS II) was designed, built, and put into operation. Specimens may be burned vertically or horizontally without the use of reflectors, to provide a uniform external radiant flux field. The flux range is 25 to 80 kW/m ² . Heat release rate, mass loss rate, smoke, and heat of combustion of the unburned gaseous decomposition products are measured. The heat release rate measurement involves the use of a substitution burner technique which provides fast response. The ratio of the heat release to mass loss rates, the apparent heat of combustion, is provided as a function of time. The calorimeter may also be operated in other modes, e.g., stack temperature increase and oxygen depletion, to obtain the heat release rate. Representative data are reported for wood and a number of synthetic polymers burned horizontally at a flux of 50 kW/m ² . These specimens generate considerable heat and a significant amount of uncombusted fuel. A program involving thorough characterization of the instrument, obtaining reference heat release data for numerous materials, and conducting research in heat release rate and other flammability characteristics is recommended.			
12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons) calorimeter; fire test; heat of combustion; heat release rate; mass loss rate.			
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